FINAL REPORT

CONTRACT 961004

"A Sensitive Magnetoresistive MEMS Acoustic Sensor"

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Introduction

This project was concerned with the development of a robust accelerometer based on modern MEMS technology and the giant magnetoresistive effect (GMR effect). The proposed device consists of a GMR sensor mounted on a suitable substrate. In close proximity to the sensor, a hard thin-film permanent magnet would be deposited on a thin membrane fabricated using MEMS (micro electromechanical systems) technology. Acceleration of the sensor would cause a deflection of the thin MEMS membrane, resulting in a change in the magnetic field near the sensor. This change in field would be detected by the spin valve and could be routed to suitable electrical circuitry. The resulting sensor would be sensitive, robust and could be fabricated very inexpensively.

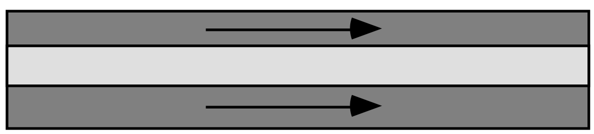
During the course of this project a GMR based sensor, a spin valve, was developed and fabricated. Spin valves based on Co/Cu/FeMn, NiFe/Cu/NiFe/FeMn and NiFe/Co/Cu/NiFe/FeMn were fabricated and evaluated. It was determined that the NiFe/Cu/NiFe/FeMn system was most suitable for this application. Although this system showed somewhat lower GMR effect than the Co based systems, it was more sensitive to small fields and was thus more appropriate for the proposed application. Several different sensors configuration were fabricated and evaluated for their performance.

The Giant Magnetoresistive Effect

The motion transducer is based on the giant magnetoresistive (GMR) effect. A GMR sensor is a multi-layer device, whose operation is based upon spin-dependent scattering of electrons, depending upon the magnetic state of the structure. The basic structure was discovered in 1991 and has been termed a "spin valve". The basic structure of the spin valve is shown in Figure 1. It consists of two ferromagnetic layers separated

Figure 1. Basic Spin Valve Structure

by a very thin non-magnetic layer. Because of shape anisotropy, the magnetization of both

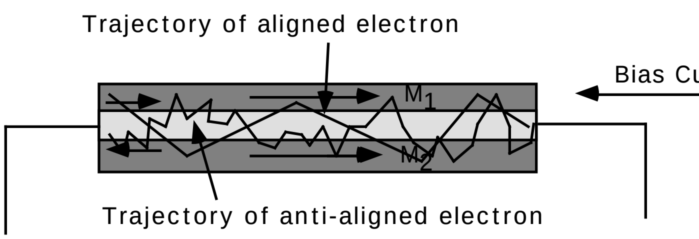


of the ferromagnetic layers lie in-plane. Usually, the magnetization in one ferromagnetic layer is fixed, or pinned along a predetermined direction. The magnetization in the other or "free" layer is allowed to rotate under the influence of an external magnetic field.

Scattering of electrons in both magnetic layers is spin-dependent. That is, electrons with their spins aligned parallel to the magnetization have quite long mean free paths, while electrons with their spins aligned anti-parallel to the magnetization have short mean free paths. This comes about from splitting of the d-bands in the material due to exchange interaction with the external field. The d-band for electrons aligned with the magnetic field is pushed down below the Fermi level, and becomes completely filled. As a result there are no allowed states into which d-band electrons can scatter, and only 4-s electrons undergo scattering. The d-band for anti-aligned electrons is pushed up such that the Fermi level runs nearly through the middle, and hence there are many empty states into which electrons can scatter, and so the mean free path is reduced.

The spin valve magnetic sensor is simply the basic spin valve device, with a bias current flowing through it. Electrons associated with the bias current flow down the device and are scattered back and forth between the two magnetic layers.

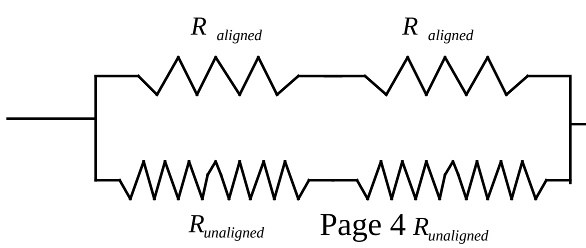
Figure 2 GMR Sensor



When the both layers have their magnetization aligned with one another, half of the electrons (with the preferred spin state) always experience very low scattering regardless of whether they are in the upper or lower magnetic layer. Hence they represent a relatively low resistance path through the sensor, which we can call $2R_{aligned}$

Electrons with their spin orientated anti-parallel to the magnetic field suffer a large number of collisions, and hence represent a relatively high resistance path, which we can call $2R_{unaligned}$.

Figure 3. Resistor representation of GMR sensor with aligned layers



These two resistors are in parallel with one another, and hence the effective resistance of the sensor is $R_{parallel} = \dfrac{2R_{aligned}R_{unaligned}}{R_{aligned} + R_{unaligned}}$.

When the magnetic fields in then two ferromagnetic layers are aligned in opposite

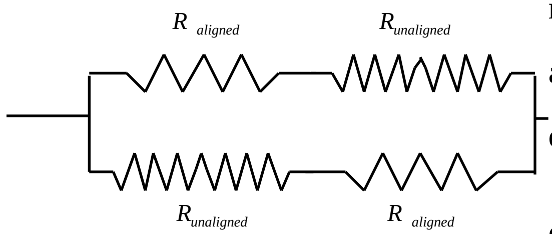


Figure 4. Resistor representation when layers are aligned in opposite directions

directions all of the electrons, regardless of their spin orientation experience a resistance of $R_{aligneed}$ and $R_{unaligned}$ as they scatter back an forth between a layer where they are aligned with the field in that layer to the other, where their spin is anti-parallel to the field in the ferromagnet.

.In this case, the resistance of the device is simply

$$R_{antiparallel} = \frac{R_{aligned} + R_{unaligned}}{2}.$$

One of the primary figures of merit for a GMR spin valve is called the GMR ratio which is simply the change in resistance of the device normalized to the unaligned resistance and is given by the expression

$$GMR \quad ratio = \frac{\Delta R}{R_{unaligned}} = \frac{(R_{aligned} - R_{unaligned})^2}{(R_{aligned} + R_{unaligned})^2}.$$

In practice, the orientation of the magnetic field in the "free" layer can take on any value from complete alignment with the pinned layer, to being aligned in the opposite direction. The resistance is given by

$$R = R_{antiparallel} + \frac{R_{antiparallel} - R_{parallel}}{2}(1 - \cos(\theta))$$

When θ is approximately $\pi/2$ the response of the GMR sensor is at its maximum, and is approximately linear for small changes in alignment. On the other hand when θ is either zero or π, the small signal response goes essentially to zero.

Fabrication Techniques

The general procedures developed for fabricating the GMR vibration detector will be outlined in this section . A detail procedure, in list format, is included at the end of this report. The GMR elements used in the vibration sensor are fabricated using physical vapor deposition by DC magnetron sputtering. The sputtering chamber which was used was made out of stainless steel and was 30" in diameter. The chamber is pumped by a 12" APB cryopump, and can achieve a minimum base pressure of about 10^{-8} torr. It is equipped with a load lock and transfer arm, so that samples may be laced in the chamber without breaking vacuum. The chamber has 5 separate ports to which sputter guns may be attached. The sample is mounted on a sample stage which can then be conveniently rotated to any particular target for deposition of the desired material. A sketch of the chamber is shown in Figure 5.

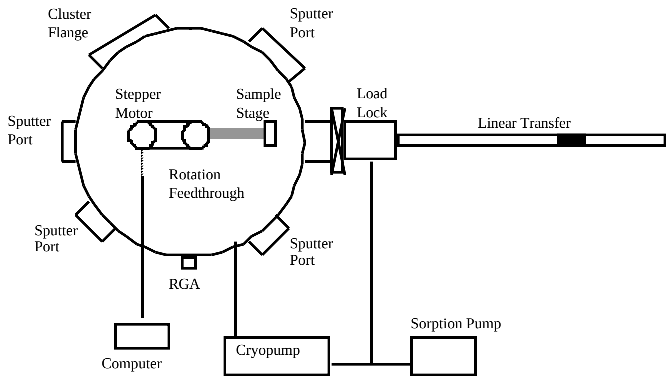


Figure 5. Deposition Chamber

The film thickness is controlled by the amount of energy delivered to the sputter gun, the argon pressure in the chamber and the sputtering time. The deposition rate for each target is determined by measuring the thickness of several calibration samples using low angle x-ray diffraction. By observing the separation of various Bragg peaks as the sample is rotated it is possible to measure the film thickness with an accuracy of only a few Å. A special holder was designed and built which permits the application of approximately 50 Oe of magnetic field bias during the deposition process in order to set the direction of magnetization of the pinned layer.

All layers were sputter deposited with a pressure of 1.0 ± 0.1 millitorr argon, with DC power of from 30 to 50 Watts applied to the 1.3" diameter MiniMak targets. In most cases the target to substrate separation was 6", and deposition rates of from 0.7 to 3.6 Å/sec were observed. Specific details are listed in the Appendix.

The most important considerations for fabrication of spin valve multi layers are cleanliness and film smoothness. Surface roughness between layers increases magnetic coupling between the two layers, and might result in unintentional pinning of the "free" layer in the spin valve. Impurities incorporated into the films during deposition results in increased electron scattering, and can degrade the GMR ratio.

Silicon wafers were chosen for the substrate material because they are quite flat and smooth and are readily available for reasonable cost. The wafers were cleaned in a dilute HF acid etch. They were then placed in an oxidation furnace, and an insulating oxide layer of several thousand Å was grown by wet oxidation in steam at 1000^o C. The oxide layer was needed to provide electrical isolation between the GMR device and the silicon substrate.

The wafers were then scribed into squares about 2 mm by 2 mm and were used as substrates in the sputter deposition system. In order to fabricate the proposed GMR sensor it is necessary to pattern the GMR film into the desired shape. Attempts to achieve this by sputtering though a shadow mask were unsuccessful, because of magnetic coupling around the rather soft and ill-defined edges of the device.

Ion milling would have provided an excellent method for defining the device shape, but this capability was not available here at Rice University. In its place, a rather sophisticated lift-off technique was developed. Prior to film deposition, photoresist is applied to the substrate and baked. The photoresist is patterned in such a way that it remains on the substrate everywhere that we do not want a final GMR film. Then, after the sputter deposition process is completed, the remaining photoresist is dissolved in acetone, lifting off the unwanted metal in the process.

The main problem with this technique is that the metal tends to deposit on the side walls of the photoresist, making lift-off somewhat difficult, and it often results in ragged edges of the device which can degrade GMR performance. A technique was developed whereby the photoresist pattern ends up being undercut after development. This then "shadows" the deposition of the metal films, resulting in much superior lift-off. The undercutting is achieved by soaking the freshly exposed pattern to chlorobenzene for about 15 minutes , followed by a post-bake.

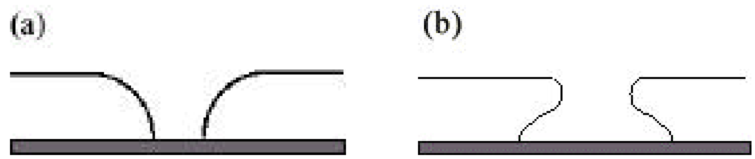


Figure 6. Regular photoresist profile and undercut resist profile

Once the GMR films had been deposited, they were characterized by measuring their magnetization characteristics in a vibrating sample magnetometer. Basically, the film is placed between the pole pieces of a well-controlled electromagnetic. The sample is attached to a rod, which is driven into vibration by a drive head. A pair of pick-up coils and a lock-in amplifier detect the voltage induced in the coils by the vibrating sample, whose amplitude is proportional to the magnetization of the sample.

Magnetic Characterization

For any ferromagnetic materials, when an external field is applied, the domains aligned along the direction of the applied field grow, while those pointing in other directions shrink. "Sticking" of domain walls introduces some hysteresis, resulting in the familiar ferromagnetic magnetization curve.

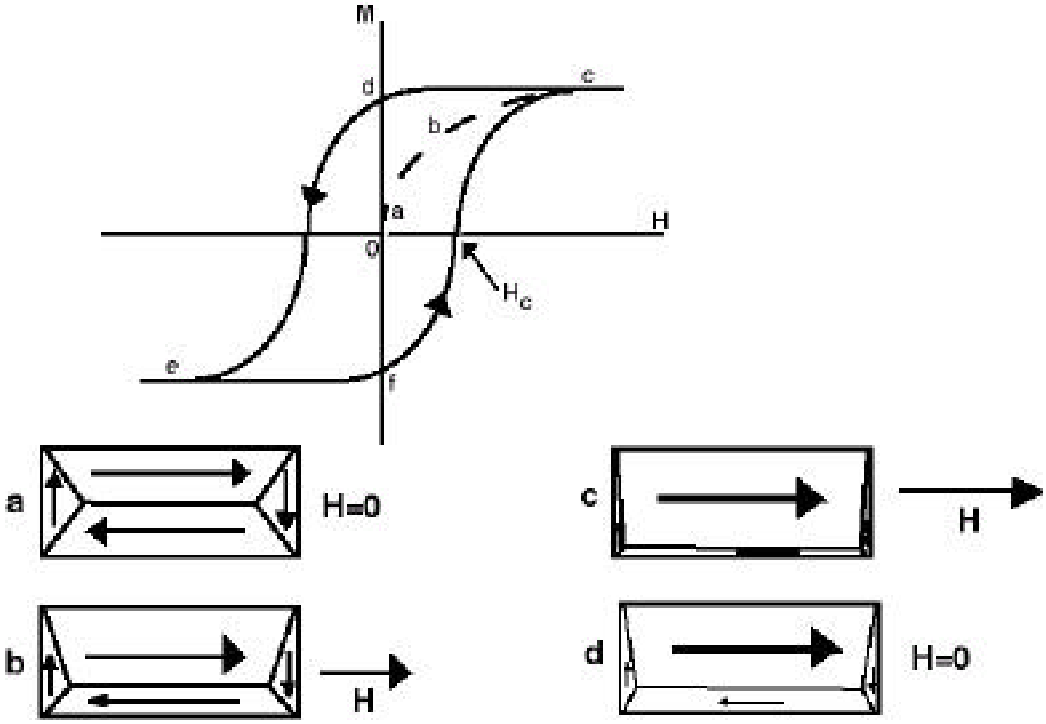


Figure 7. Domain growth an magnetization curve.

For a virgin sample, starting at point (a), the application of an applied field grows the favored domain until it almost entirely fills the sample, at which point (c) we say that it is saturated. The value of M at this point is called the saturation magnetization, or M_s. As the applied field is reduced back to zero, the domains relax somewhat, but do not go back to their original state, and hence there is some remanence magnetization as at (d). A reverse filed, called the coercivity field H_C is required to bring the magnetization back down to zero.

If a material such as Permalloy is deposited in the presence of an applied external field, there will be built into the material a preferred direction of magnetization. This is called anisotropy. The preferred direction is called the "easy axis" of magnetization, while the perpendicular direction is called the "hard axis". If, after the film has been deposited, an external field is applied along the easy axis the magnetization will occur primarily by domain wall motion, and an open hysteresis loop is obtained. If the external field is applied along the hard axis, magnetization occurs primarily by rotation, and a more or less linear M-H loop is obtained.

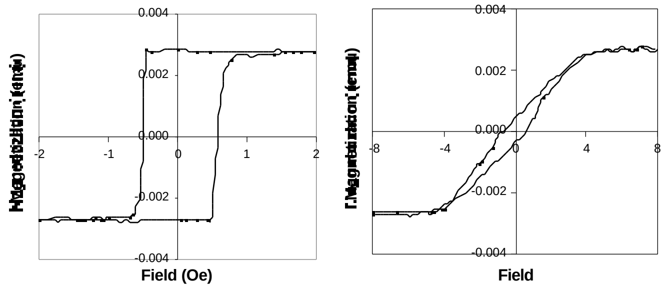


Figure 8. Easy and hard axis M-H loops for a Permalloy film.

In the spin valves reported here, one of the two ferromagnetic layers is pinned in a preferred direction by coupling it to an adjacent antiferromagnetic layer. By depositing the antiferromagnetic layer in the presence of an external field, the direction of its magnetization is set, which then "pins" the magnetization of the neighboring ferromagnetic layer. A non-magnetic layer is deposited between the pinned ferromagnetic layer and the "free" layer. The magnetization of the free layer is coupled to the pinned through the non-magnetic layer by Néel coupling, which comes from the non-planarity of the film structure.

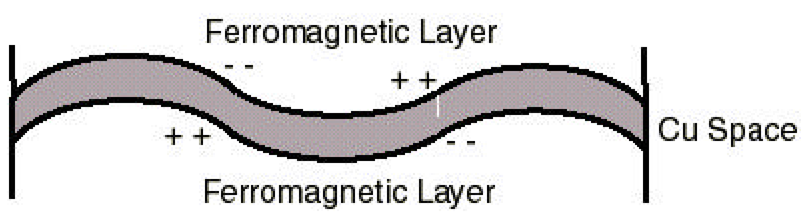
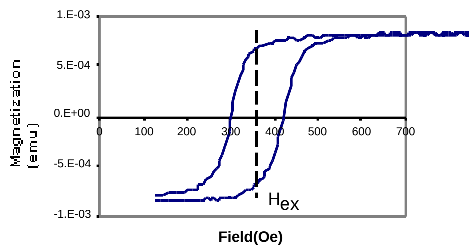


Figure 9. Néel coupling between two ferromagnetic layers

This coupling between the pinned and free layers results in an off-set of the M-H loop of the free layer by and amount called H_{ex}, the exchange field. The exchange field is a direct measurement of the exchange coupling across the non-magnetic interface. In a spin valve the pinned and free layers are usually fairly heavily coupled, and is strongly influenced by the thickness of the non-magnetic layer.

Figure 10. M-H loop of a free layer couple to a pinned layer



Spin Valve Overview and Characterization

The basic structure of a complete spin-valve device is shown in Figure 11.

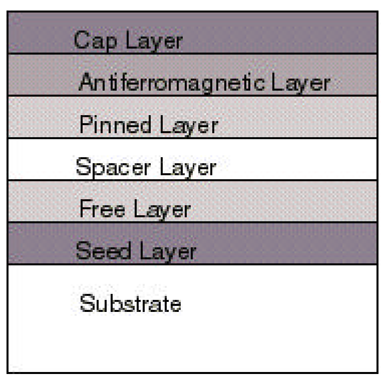


Figure 11. Spin valve structure.

The substrate is the silicon dioxide surface of the oxidized silicon wafer. Tantalum is first applied as a seed layer. It provides for a very smooth surface, and also promotes adhesion of the subsequent magnetic layers. Cobalt, Permalloy or an alloy of nickle, cobalt anf iron are usually chosen for the free and pinned ferromagnetic layers. Permalloy is the softest (lowest coercivity) material and is chosen for low field applications. Permalloy

GMR spin valves achieve GMR ratios on the order of 5%. Copper is often used as the spacer layer, as it is non-magnetic yet still provides a low-scattering communication for electrons from one magnetic layer to the other. Finally an additional capping layer of Tantalum is added to protect the GMR device from corrosion. The films used in this work were Peramalloy-based, with an iron-manganese antiferromagnetic layer. The spacer layer was copper.

There are several choices for orientation of both the anisotropy direction in the free layer, as well as the applied external field for a GMR sensor. One approach is to make the easy axis of the free layer aligned with the direction of the pinned layer. This results in a large GRM ratio, but shows a large amount of hysteresis in the R-vs-H curve which makes it undesirable for device applications.

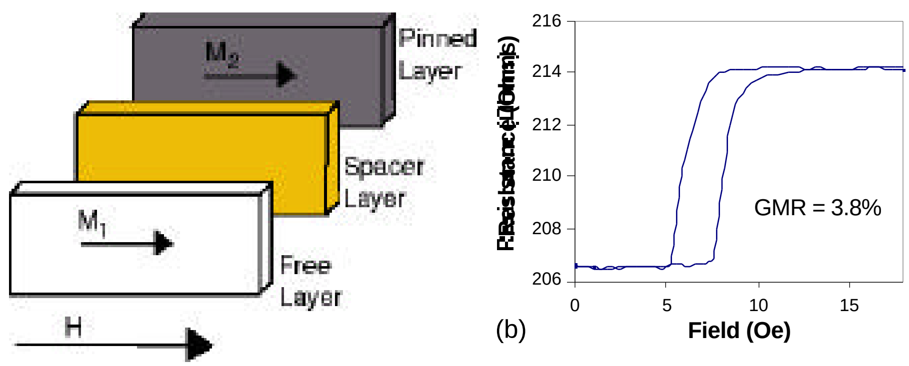


Figure 12. Pinned layer, easy axis and applied field all along same direction.

An alternative approach is to align the easy axis of the free layer with that of the pinned layer, but apply the external magnetic field in the perpendicular direction.

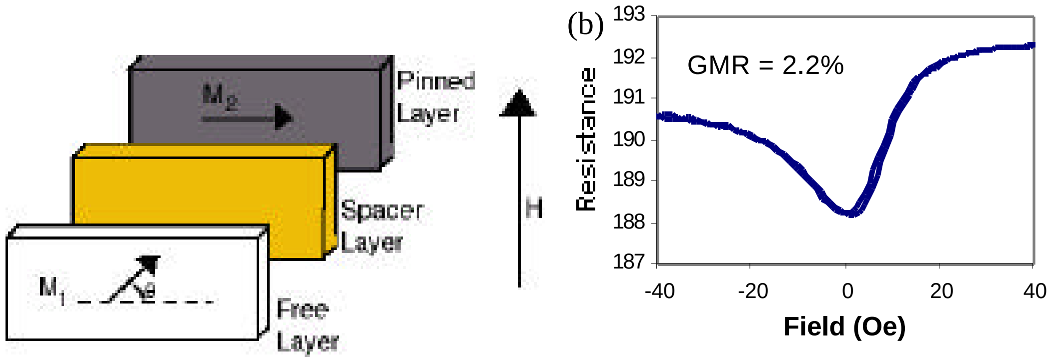


Figure 13. Applied field perpendicular to the pinned layer and easy axis.

The change is resistance is less for this configuration, since the alignment of the free layer never gets more than ± 90° from the direction of the pinned layer.

The best sensing configuration for a simple spin valve is referred to as the "crossed easy axis" configuration. In this approach, the pinning direction is perpendicular to the easy axis of the free layer, and the applied field is also aligned perpendicular to the free layer easy axis.

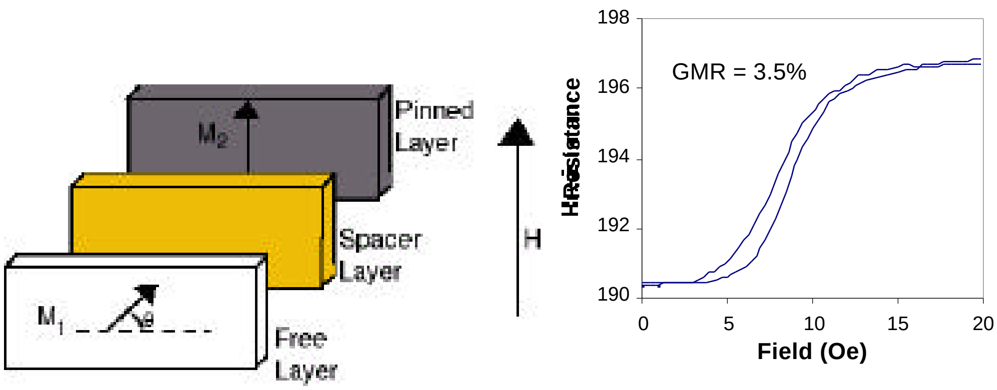


Figure 14. Crossed easy axis configuration.

The GMR ratio is again near its maximum value, and the response is linear and relatively hysteresis free. In order to achieve this configuration, the field applied during deposition must be rotated 90° between the deposition of the free layer and the antiferromagnetic pinning layer, or the device must be heated above the Néel temperature of the antiferromagnetic layer, and an external rotating field applied to the device.

The thickness of the copper spacer layer is critical to good device performance. It must be thin enough so that electrons can pass though without scattering, yet it must be thick enough so that the free layer is not too strongly coupled to the pinned layer, in order that it may respond to an external magnetic field. . Figure 15 shows the dependence of GMR ration on copper thickness.

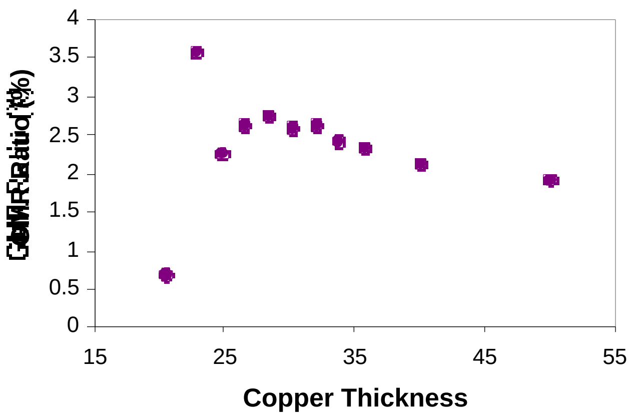


Figure 15. Dependence of GMR ratio on copper spacer layer thickness

If the copper is too thin (below 20 Å or so) the two magnetic layers are coupled together, and hence very little GMR ratio is seen. As the copper gets thicker, electron scattering as well as conductive shunting by the copper film itself lower the GMR ratio.

The thickness of the Permalloy ferromagnetic layers can also influence device performance. This is primarily through changes in Permalloy grain size. As seen in Figure 16, shows the results of X-ray studies of grain size as a function of film thickness.

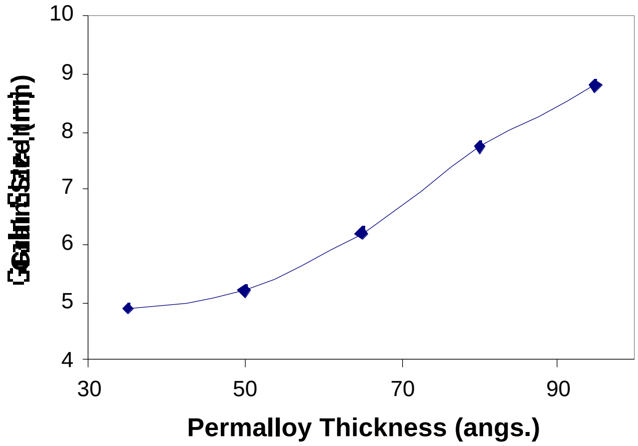


Figure 16. Grain size as a function of Permalloy thickness

Because of increased surface coverage and lower defect density (larger grain size) there is a pronounced minimum in coercivity field at around 80 Å of thickness. This result is in good agreement with observations made by other workers in the field

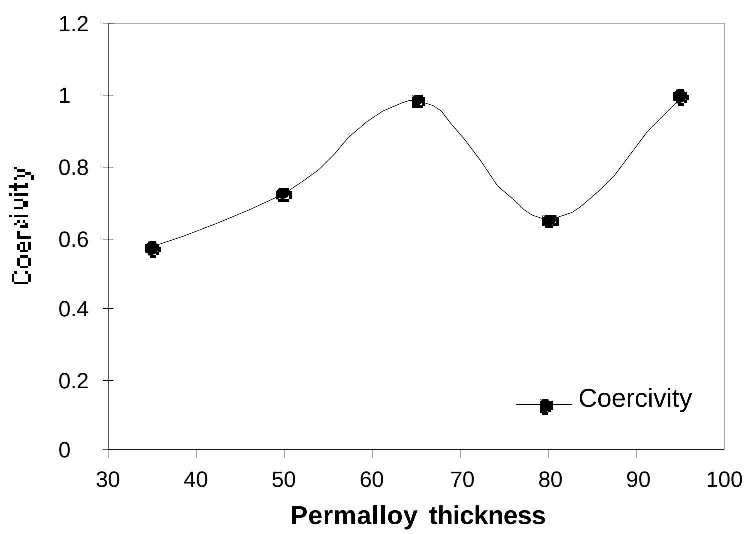


Figure 17 Coercivity of Permalloy film as a function of thickness.

The GMR ratio is also a function of Permalloy film thickness. For films that are too thin, electron scattering at the film interfaces begins to dominate the resistance. Since interface scattering is not spin dependent, it shows no GMR effect. If the Permalloy film becomes too thick, its overall resistance begins to decrease, and in effect short out the GMR effect.

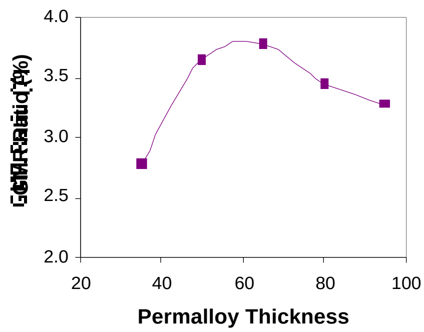


Figure 18. GMR ratio as a function of Permalloy thickness.

As can be seen in Figure 18, the optimum Permalloy thickness is between 50 and 75 Å.

The magnetic coupling between the free and pinned layers is also affected by the thickness of the Permalloy layer. For thin layers, it decreases roughly as 1/t, where t is the film thickness. This is because as the film gets thicker, the moment arm for rotations also increase and the magnetization becomes more continuous. For thicker films however, the grain size, and hence the roughness increase, and the Néel coupling between the layers increases. The ferromagnetic coupling between the layers is also proportional to their thickness which leads to a greater coupling field. These effects are seen in Figure 19.

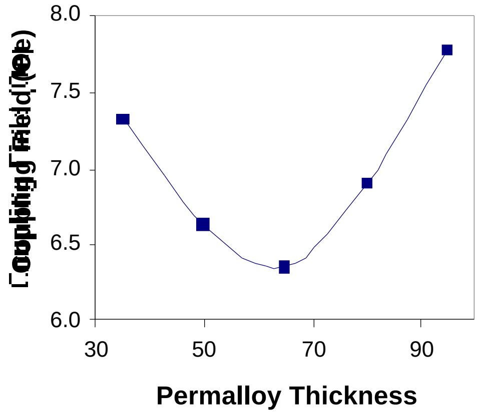


Figure 19. Inter-level coupling field as a function of Permalloy thickness.

Finally, the argon pressure in the sputtering chamber during deposition can effect the sensitivity of the film. Increasing argon pressure results in rougher films, and hence greater Néel coupling through the non-magnetic copper layer. This increased coupling makes it more difficult to rotate the free layer, and hence reduces the GMR ratio. This is shown in Figure 20, which plots the response of a set of Ta 50/Py 50/Cu 35/Py 50/FeMn 120/Ta 50 devices to an applied field for various argon sputtering pressures.

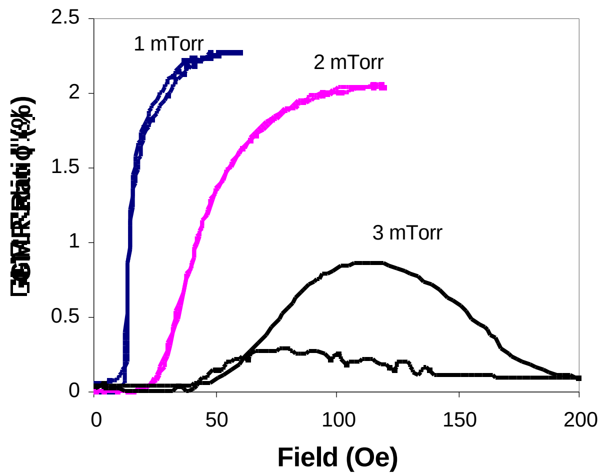


Figure 20. GMR response for films sputtered at different argon pressure.

Also as part of this work, a number of studies were made on the noise characteristics of the GMR spin valve devices. A special probe station was constructed with a custom electromagnet which could be used to both bias the spin valve as well as apply a small ac signal field. The magnet was machined from HyMu alloy and with the use of a programmable power supply, generate fields up to 200 Oe (which was limited by the saturation of the HyMu alloy) A six probe measurement set-up was also constructed.

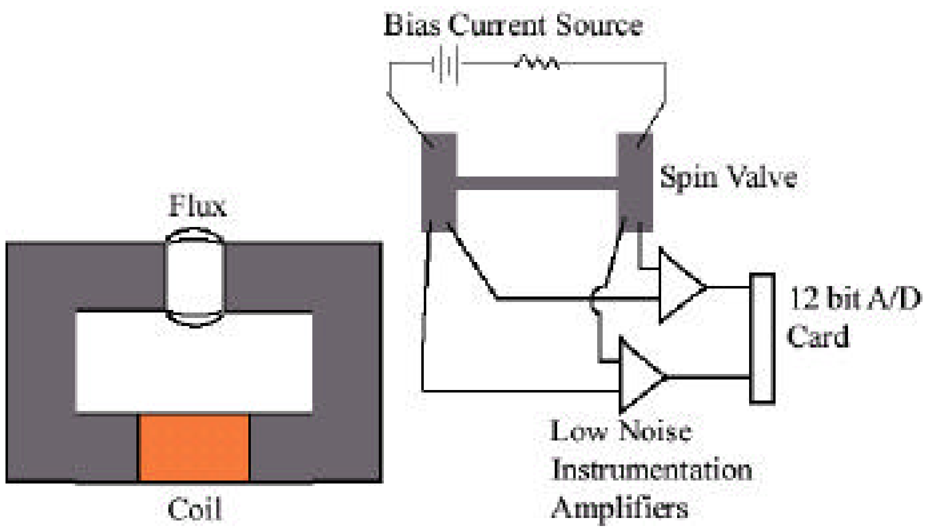


Figure 21. Experimental set up for noise studies

One pair of leads was used to provide bias current to the spin valve. The other four probes were used to measure the noise voltage across the device. The two sets of probes were amplified independently, using very low noise instrumentation amplifiers, and the resultant data stored in a computer. Taking a cross correlation of the two sets of data removes the (uncorrelated) noise from the instrumentation amplifiers, leaving only the intrinsic noise of the GMR spin valve.

The low frequency noise spectrum from a typical Permalloy/copper/Permalloy GMR spin valve is shown in Figure 22.

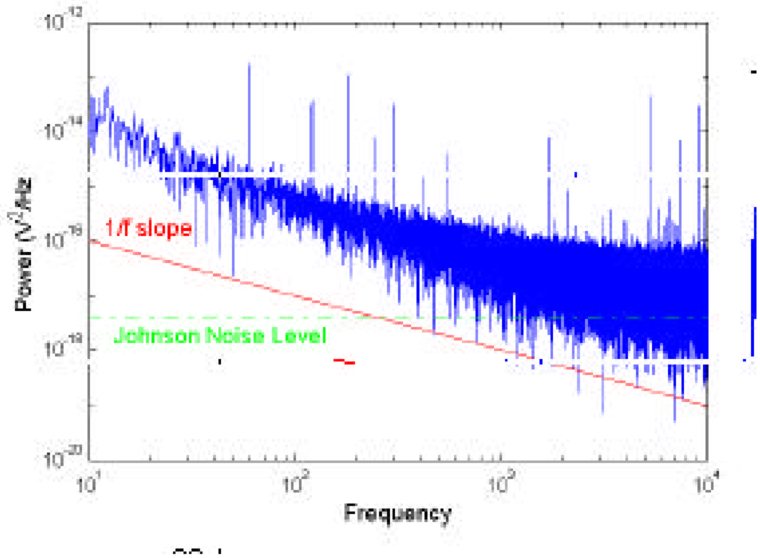


Figure 22. Low frequency noise spectrum of GMR spin valve device.

These characteristics are important for they will determine the performance limit of any potential GMR based accelerometer. As can be seen in Figure 21, The low frequency noise exceeds the resistive Johnson noise by about 2 orders of magnitude, and displays a characteristic 1/f frequency dependence.

Typical response of the noise power as a function of applied magnetic field for a crossed easy axis device is shown in Figure 23.

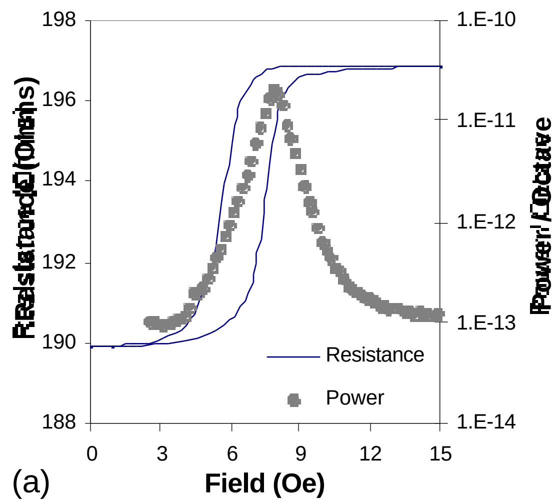


Figure 23. Noise power as a function of bias field for a crossed easy axis GMR spin valve.

Since the noise power correlates directly with the sensitivity of the GMR device (as indicated by the slope of the resistance v. field curve) it is clear that the noise is a direct result of the GMR effect, and not due to contacts or other related phenomena. We did find some indication that higher quality films exhibited less noise than lower quality ones, and so it may be possible to use noise characterization as a means for judging the quality of a GMR spin valve.

GMR Spin Valve Based Accelerometer Construction

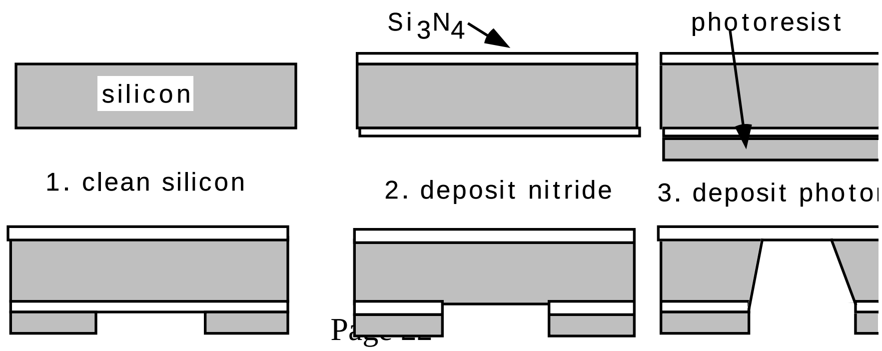
The initial design for the spin valve based sensor is shown below in Figure 24.

Fi

An ch had been treated wi ttern. The substrate f Ta, NiFe (Permallo ved from the sputtering in valve material. ow mask to define their placement.

The micromachined silicon nitride membranes were prepared for JPL by an outside vendor and supplied to us. The nitride membranes were 0.5 μm thick with die sizes from 2mm square up to 5 mm square. The larger membranes were quite fragile, and often broke during processing. After a potassium hydroxide soak to remove silicon dust, they were rinsed in DI water and baked dry.

A hard $Co_{0.7}Pt_{.3}$ layer was deposited on silicon nitride membrane through a shadow mask. The shadow masks were fabricated from silicon wafers, also using MEMS



technology. First a silicon nitride layer was deposited on both side of the silicon by reactively sputtering silicon in a 10% N_2 in argon atmosphere. One side was then patterned with photoresist and etched by plasma etching (using PolyEtch gas) to open a window in the silicon nitride. The photoresist is removed, and the silicon is etched in a 30% KOH solution at 80^o C. The etch takes about 4 hours to completely go through the silicon, etching preferentially along the <111> direction. The process steps are illustrated in Figure 25 below.

Figure 25. MEMS process steps for making a shadow mask or sensor membrane.

Figure 26 shows a M-H loop for one of the hard CoPt permanent magnet layers.

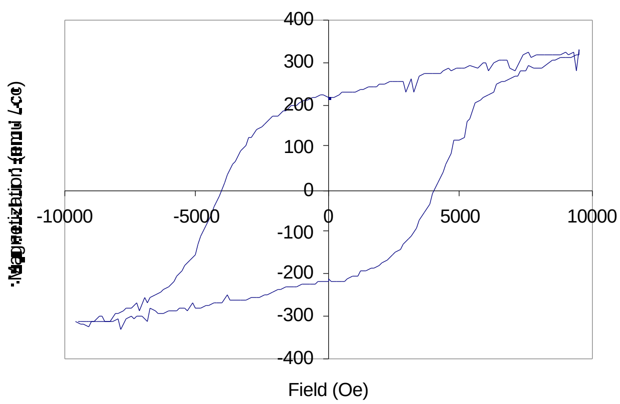


Figure 26. Magnetic Characterization of the CoPt thin film.

The difficulty with this sensor design turned out to be in the bonding process between the substrate containing the GMR spin valve and the MEMS membrane which supported the CoPt magnet. We tried several materials for the spacer layer; silicon nitride, silicon dioxide and photoresist. The nitride could be patterned by masking during deposition. The oxide was hard to etch because of masking problems, and was not used in the end. The photoresist was relatively straightforward to use. The magnetic sensor and magnet/membrane were pressed together and epoxy bonded. A figure of a completed device is shown in Figure 27.

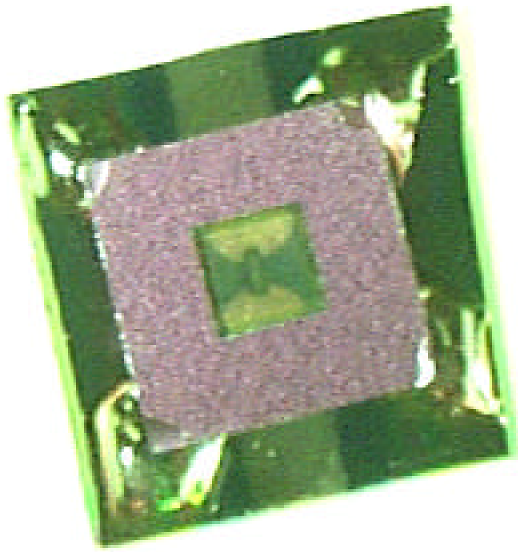


Figure 27. Photo of initial device prototype. Overall device is about 1.5 cm. square. The GMR sensor and contacting copper leads can be seen in the center under the membrane

Unfortunately, we were not able to observe any vibration response from the devices which were fabricated. It is now believed that the fringing fields from the bar design magnet were too non-uniform to cause more than just a small portion of the spin valve to switch at any given position.

In light of the lack of response of the initial accelerometer design, a second design and fabrication process was devised. This design is based on placing the sensor on the membrane, rather than the permanent magnet, and then placing the resultant device in a strong magnetic field gradient created by a pair of external magnets. The basic process steps are outlined in Figure 28

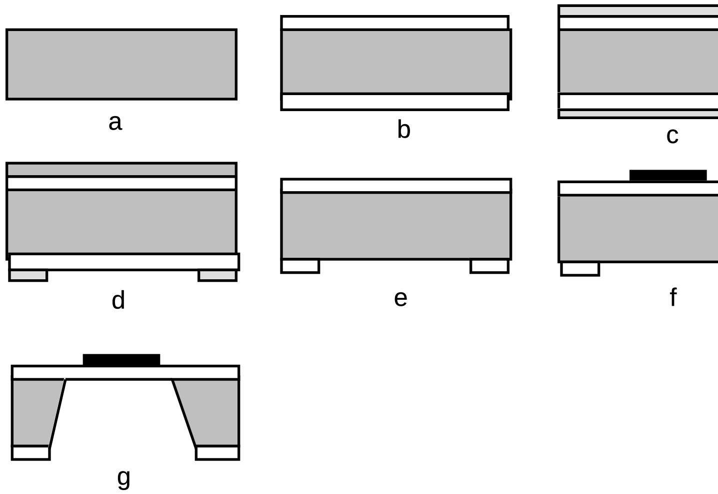


Figure 28. Proposed new process for making MEMS accelerometer.

Staring with a <100> silicon wafer (a), have low stress silicon nitride deposited on both sides (b). Deposit photoresist on both sides of the wafer (c), expose and develop one side of the wafer to create a window (d). Using a plasma etching, etch away the silicon nitride on that side (e). Using the standard techniques, deposit a GMR device on the top side of the wafer. Using a special protective holder (shown in Figure 29.) protect the top side of the wafer while the bottom is immersed in KOH to etch away the silicon, leaving the GRM sensor suspended on the nitride membrane.

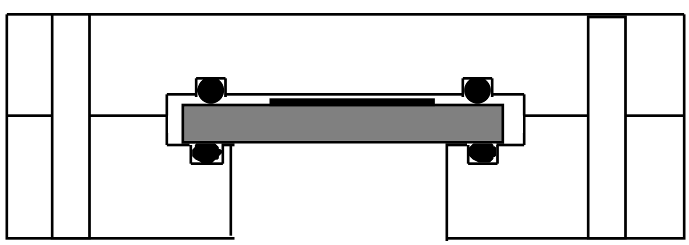


Figure 29. Sample holder for etching.

The holder is constructed of Teflon and is designed to protect the top layer containing the GMR sensor while the silicon is being etched to free the nitride membrane.

Once completed, the device can be mounted in between two permanent magnets who have similar poles facing one another. The creates a very strong magnetic field gradient in the region around the GMR sensor, and also makes using the crossed easy axis configuration quite straightforward. Altering the bias field on the device is also greatly simplified, since it only needs to be moved back and forth in between the two permanent magnets. Unfortunately, limitations in time, personnel and funding did not allow us to completely work out all of the issues in developing this process before the research period ended.

Appendix: Specific process steps for fabricating GMR spin valve sensors.

1. Etch silicon wafers in HF to clean off residual oxide.

2. Oxidize wafers at 950^{o} C in oxygen for 2-3 hours

3. Scribe wafers into suitable size substrates

4. Pre-bake to dry substrate at > 150^{o} C for 30 minutes

5. Apply Shipley 1813 photoresist and softbake at 90^{o} C for 30 min.

6. Expose photoresist to inverse of spin valve pattern

7. Soak substrate in chlorobenzene for 15 minutes

8. Bake at 80^{0} C for 15 minutes

9. Develop resist for 2 minutes in Shipley MF-20 developer.

10. Place substrate in sputter apparatus and deposit spin valve. (Times and power will vary with the specific apparatus used, which will have to be calibrated.). One millitorr of argon pressure is recommended for the sputtering pressure. For our system the following was used:

Target Material	Power Watts	Ar Pressure millitorr	Separation inches	Deposition Rate Å/sec
Tantalum	50	1	6	0.77
Permalloy	50	1	6	0.72
Copper	30	1	6	1.23
Iron Manganese	30	1	3	3.63
Cobalt	50	1	6	0.86

11. Typical layer thicknesses were: Ta 50Å, NiFe 75Å, Cu 27 Å, NiFe , NiFe 50 Å, FeMn 120 Å, Ta 50Å. Typical device dimensions were 150 μm wide by 900 μm long.

12. Remove substrate, mount shadow ask and return to sputter chamber.

13. Sputter on copper contacts.